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(54) ELASTOMERIC LATEX AND METHOD OF PREPARATION THEREOF

(71) We, JEFFERSON CHEMICAL COMPANY, INC., a Corporation organized under laws of the State of Delaware, United States of America, of 3336 Richmond Avenue, P.O. Box 53300, Houston, State of Texas, United States of America, do hereby declare the invention, for which we pray that a Patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to film forming urethane latices and the preparation thereof. It more particularly relates to urethane latices which are stable oil-in-water emulsions and which form films with outstanding physical properties.

In general, urethane latices are known in the art. However, in many instances these latices fail to form a film or coating having satisfactory physical properties or require a high temperature curing or fusion step in order to maximize physical properties. In many potential latex applications, fusion would be an undesirable, if not impossible, final step. When cast to form a film, some latices crack (commonly called "mud cracking") upon drying. Other coagulate and fail to form a stable emulsion upon addition of known chain-extending agents to the prepolymer emulsion.

Various polyurethane latices are discussed in U.S. Patent No. 2,968,575 where primary diamines such as aromatic and aliphatic diamines are used as the chain-extending agent. U.S. Patent No. 3,294,724 discusses the production of film-forming urethane latices using an alkylpiperazine as the chain-extending agent. While the films prepared by the process taught in the above-mentioned patents are satisfactory in some respects, much is left to be desired where the versatility of the chain-extending agent is concerned, both as to forming the latex and producing the film.

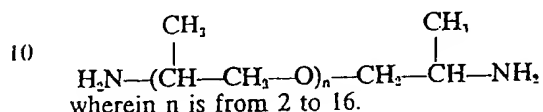
In the production of polyurethane latices, many variables have been found which materially affect the quality of film produced—if, in fact, any film is produced at all. These factors also become relevant when determining whether or not the emulsion of the polyurethane latex is stable in the water. Some of the variables which must be considered are the particular polyols and diisocyanates which combine to produce the isocyanato-terminated prepolymer. Also, in this regard the ratio of isocyanato to hydroxyl groups which go into producing the prepolymer must be considered. An additional factor is whether or not a solvent is necessary for the isocyanato-terminated prepolymer to be emulsified in the aqueous medium which acts as a carrier for the polyurethane particles once they have been formed.

Another aspect of the formation of polyurethane latices is the type and amount of emulsifier which is used to form the oil and water emulsion of the reaction product. In addition, mixing used in the formation of the prepolymer and the chain-extending reaction has also been found to be of importance. A discussion of the above parameters and variables is found in the aforementioned patents. However, the chain-extending agents used therein have been found wanting in the variety of combinations of parameters available in the formation of the urethane latices.

According to the present invention there is provided an elastomeric latex comprising an oil-in-water emulsion of the reaction product of:

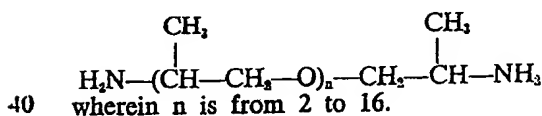
- (a) an isocyanato-terminated prepolymer prepared by reacting a molar excess of an organic diisocyanate and a polymeric diol having a molecular weight of from 400 to 2,000, the molar ratio of the diisocyanate to the polymeric diol being between 1.5:1 and 2.0:1 and,

- (b) 0.8 to 1.2 molar equivalents of a polyoxypolylenediamine chain extender per molar equivalent of excess isocyanato groups of the prepolymer, said polyoxypolylenediamine having an average molecular weight of from 190 to 1,000 and having the general formula:



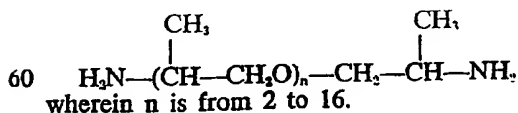
- Also according to the present invention there is provided a process for the manufacture of stable elastomeric latex which comprises:

- (a) reacting a molar excess of an organic diisocyanate with a polymeric diol having a molecular weight of from 400 to 2,000, the molar ratio of the diisocyanate to the polymeric diol being between 1.5:1 and 2.0:1, to form an isocyanato terminated prepolymer;
- (b) emulsifying the prepolymer in an aqueous medium with agitation in the presence of an emulsifying agent which will give oil-in-water emulsion, and
- (c) chain extending the emulsified prepolymer by adding 0.8 to 1.2 molar equivalents per molar equivalent unreacted isocyanato group of a polyoxypolylenediamine having a molecular weight of from 190 to 1,000 and of the general formula:



- When cast as films the latices of this invention give a resilient film with desirable physical properties such that a high temperature curing, or fusion step, is not necessary. In many applications for such films it is inconvenient, if not impossible, to provide such fusion as, for example, when the film is used to seal a concrete flooring surface.

- The chain-extending agents which may be employed in accordance with this invention are those polyoxypolylenediamines having a molecular weight of from 190 to 1,000. These polyoxypolylenediamine chain extenders may be more particularly described by the following formula:



- Preferred polyoxypolylenediamines are those having a molecular weight of from 190 to 500, or, n is 2 to 7. Particularly preferred

polyoxypolylenediamines have a molecular weight of from 190 to 400, or, n is 2 to 5.5. The amount of the polyoxypolylenediamine to be used in the chain-extension step is such that from 0.8 to 1.2 equivalents of the polyoxypolylenediamine is present in the chain extension reaction for each unreacted isocyanato equivalent in the isocyanato-terminated polyurethane prepolymer. It is especially preferred that 1.0 equivalent of the polyoxypolylenediamine is present for each unreacted isocyanato equivalent in the prepolymer.

The polyoxypolylenediamines useful in the practice of this invention are prepared by any of the known methods for preparing such compounds. For example, U.S. Patent No. 3,236,895 describes a method of preparing the polyoxypolylenediamine from the corresponding polyoxypolyene glycol. These diamines are prepared by the reaction of ammonia with certain esters of the corresponding polyoxyalkylene glycol or by treatment of the corresponding polyoxyalkylene glycol with ammonia and hydrogen in the presence of a Raney nickel catalyst. Another method of preparing these compounds is described in British Patent No. 1,185,239, wherein the corresponding polyoxypolyene glycol is reacted with hydrogen and ammonia in the presence of a catalyst prepared by the reduction with hydrogen of a mixture of the oxides of nickel, copper and chromium.

The polyoxypolylenediamine may be added directly or in the form of an aqueous solution thereof. Preferably, the chain extender is added in an aqueous solution. The emulsifying agent may be added either to the prepolymer or to the aqueous medium in which the prepolymer is to be emulsified, or may be formed in situ during addition of the prepolymer to the said medium.

The polyoxypolylenediamine chain-extending agent utilized in accordance with this invention may be used in the form of aqueous solutions in producing these emulsions, or latices, since they react more readily with the isocyanate-terminated polyurethane prepolymer than does water itself. For the same reason, the prepolymer may be emulsified in water just prior to adding the chain-extending agent. A hydrogen atom on each end of the molecule of the chain-extending agent reacts preferentially with the free isocyanate groups remaining in the initial polyurethane reaction product much more readily than does the hydrogen of the water, and therefore the chain is extended by reaction with the polyoxypolylenediamine compound even though the reaction takes place in an aqueous medium.

The linear polyurethane prepolymers are prepared by the reaction, in an equivalent ratio of NCO/OH of between 1.5:1 to 2:1,

of an organic diisocyanate with a polymeric diol having an average molecular weight of from 400 to 2,000.

The linear prepolymers which may be chain extended according to this invention are isocyanato-terminated prepolymers prepared by the reaction of selected diols with an excess of organic diisocyanates. Any of a wide variety of organic diisocyanates may be employed in this reaction, including aromatic, aliphatic and cycloaliphatic diisocyanates, and combinations thereof. Representative compounds include aromatic diisocyanates such as 2,4-tolylene diisocyanate, mixtures thereof with 2,6-tolylene diisocyanate (usually about 80/20), 4,4'-methylenebis(phenylisocyanate) and m-phenylene diisocyanate. Aliphatic diisocyanates such as tetramethylene diisocyanate and hexamethylene diisocyanate, and alicyclic compounds such as 1,4-cyclohexylene diisocyanates and 4,4'-methylenebis(cyclohexylisocyanate) are also operable.

The diisocyanates may contain other substituents, although those which are free from reactive groups other than the two isocyanate groups are ordinarily preferred. In the case of the aromatic compounds, the isocyanate groups may be attached either to the same or to different rings. Additional diisocyanates which may be employed, for example, include p,p'-diphenylmethane diisocyanate, 3,3'-dimethyl-4,4'-biphenylene diisocyanate, 3,3'-dimethoxy-4,4'-biphenylene diisocyanate, 4,4'-biphenylene diisocyanate, 4-chloro-1,3-phenylene diisocyanate, 3,3'-dichloro-4,4'-biphenylene diisocyanate, and 1,5-naphthalene diisocyanate, and other diisocyanates in a blocked or semi-inactive form such as the bis-phenylcarbarnates of tolylene diisocyanate, p,p'-diphenylmethane diisocyanate, p-phenylene diisocyanate, 1,5-naphthalene diisocyanate and 1,5-tetrahydronaphthalene diisocyanate. Hydrogenated 4,4'-diphenylmethane diisocyanates sold under the trademark of **HYLENE(R)** w is particularly desirable.

The polymeric diols which may be employed in the preparation of the linear prepolymers utilized in this invention are those diols having an average molecular weight of from 400 to 2,000. It is important that the hydroxyl-containing component of the prepolymers utilized in this invention be a polymeric diol since hydroxyl-containing components other than diols may introduce excessive cross-linking into the prepolymer chain and thus films cast from latices prepared from the prepolymers would suffer from mud cracking and corresponding loss of strength properties. It is also important that the polymeric diols utilized in this invention have average molecular weights of from 400 to 2,000. At average molecular weights above 2,000, the strength of the films cast from latices prepared therewith begins

to fall off considerably while at average molecular weights below 400 the latices are difficult to prepare and films cast therefrom have poor low-temperature flexibility. The preferred polymeric diols of this invention are those which have average molecular weights between 600 and 1,200. Examples of this group include certain polyoxyalkylene glycols; or polyalkylene ether glycols, such as poly-1,2-oxypropylene glycol, poly-1,2-oxybutylene glycol, poly-1,3-oxybutylene glycol, polyoxyethylene poly-1,2-oxypropylene glycol, polyoxyethylene poly-1,2-oxybutylene glycol, polyoxyethylene poly-1,3-oxybutylene glycol, poly-1,2-oxypropylene poly-1,2-oxybutylene glycol, poly-1,2-oxypropylene poly-1,3-oxybutylene glycol, polytetramethylene ether glycol, polytrimethylene ether glycol, certain polyalkylene arylene ether glycols such as the 1,2-proplene oxide addition products of Bisphenol A, 1,2-propylene oxide addition products of xylene diol; polyalkylene ether-polythio ether glycols, and alkylene oxide adducts of primary, secondary and tertiary aliphatic, cycloaliphatic, heterocyclic and aromatic amines such as the 1,2-propylene oxide adducts of ethylamine, ethylenediamine, piperazine, 2-methylpiperazine, aniline and phenylene diamines. Preferred diols are the polyoxyalkylene glycols, particularly poly-1,2-oxypropylene glycol.

In the preparation of the linear prepolymers which are utilized in this invention, the organic diisocyanate and the polymeric diol must be employed in amounts so that the NCO/OH equivalent ratio is between 1.50:1 to 2:1. At NCO/OH equivalent ratios greater than 2:1 or less than 1.50:1 film-forming latices are either not obtained or, if obtained, upon casting, the resulting films do not possess satisfactory mechanical strength properties.

The linear prepolymer is ordinarily prepared by heating the polymeric diol and the diisocyanate, with agitation, at a temperature of from 50° C. to 130° C., preferably 70° to 100° C. If a catalyst is employed, temperatures of from 0° C. to 60° C. are satisfactory. The reactants are heated for a period sufficient to react all the hydroxy groups whereafter the prepolymer is allowed to stand and the free NCO content determined. Generally, total reaction time will be from a period of from about two hours to about two days when a catalyst is not employed and a period of from about ten minutes to about three hours with a catalyst.

The reaction is preferably carried out, but not necessarily, in the presence of a solvent. If the prepolymer is a fluid at processing temperatures, it is possible to carry out the reaction in the absence of a solvent. Convenient solvents are organic solvents having a boiling range above 90° C. when the reaction in the absence of a solvent. Con-

Lower boiling solvents may, of course, be used where the reaction is carried out in closed equipment to prevent boiling off the solvent at the temperatures of the reaction.

- 5 The solvent, when used, may be added at the beginning, at an intermediate point, or at the end of the prepolymer stage, or after cooling of the formed prepolymer.

The solvents to be used are preferably those in which the reactants are soluble. Ketones, tertiary alcohols and esters may be used. The aliphatic hydrocarbon solvents such as the heptanes, octanes and nonanes, or mixtures of such hydrocarbons obtained from naturally occurring petroleum sources such as kerosene, or from synthetically prepared hydrocarbons, may sometimes be employed. Cycloaliphatic hydrocarbons such as methylcyclohexane and aromatic hydrocarbons such as toluene may likewise be used. Hydrocarbon solvents such as toluene and benzene are preferred. The amount of solvent used may be varied widely. From about 2 to about 30 parts or even more of solvent per 100 parts of prepolymer have been found to be operable. The excess solvent, where larger amounts are employed, may be separated partially or completely from the polymer prior to emulsification in the water solution. Sometimes the excess solvent is useful and is allowed to remain during the emulsification stage.

The amount of water to be employed in the formation of the emulsion is not critical. When too small an amount of water is employed, emulsions are obtained which are too thick to handle readily while, on the other hand, dispersions which are too dilute are uneconomical to handle due to their excessive volume. It is preferred that a solution having about 50% solids results after the chain-extending reaction is complete.

Any emulsifying agent which will give oil-in-water emulsions is satisfactory for use in the present invention. Satisfactory types of emulsifying agents are the polyethylene glycol ethers of long chain alcohols, quarternary ammonium salts, the tertiary amine or alkylol amine salts of long chain alkyl acid sulphate esters, alkyl sulphonic acids or alkyl aryl sulphonic acids or salts thereof, and alkali metal salts of high molecular weight organic acids. Nonionic agents such as polyoxyethylene-polyoxypropylene glycols are preferred. The pH can then be regulated to a neutral value, preferably not above 7, to minimize any tendency toward hydrolysis. Salts of the high molecular weight organic acids may be used as emulsifying agents. One method of incorporating such salts is to mix the acid, e.g., tall oil, with the prepolymer mass and to have the requisite amount of alkali present in the aqueous bath, so as to form the emulsifier in situ. Although there is presumably some reaction between the acid and the free isocyanate groups in the prepolymer, this is not signifi-

cant if the mixture is fairly promptly added to the aqueous bath. From 2% to 6% of the emulsifying agent based on the weight of the prepolymer employed, will usually be found sufficient to produce stable emulsions. When a fatty acid soap is used as the emulsifying agent, the soap must not be destroyed by acid substances. The pH must, therefore, be maintained at least as high as that of an aqueous solution of the soap if stable latices are to be produced. For most fatty acid soaps, the pH should be at least 9, and for this reason soaps are not preferred. The small amount of carbon dioxide which may be formed by the chain extension of the isocyanate groups with water is acidic and uses up free alkali in the latex, so that an excess of alkali may be necessary to compensate for this. Preferably no alkali is added to the reaction, since some usually remains and causes deterioration of the polymer at elevated temperatures.

The chain-extension step, while a relatively fast reaction when employing the chain extenders of this invention, may frequently be assisted by agitation of the emulsion for some time after its initial formation. This is usually accomplished by means of a conventional paddle type agitator at 30 to 90 r.p.m. or other conventional stirring equipment such as a Cowles dissolver, which aids in contacting the emulsion droplets with the chain extender.

The latex formed in the practice of this invention may be useful for the casting of films on articles or either by dipping an article to be coated into the emulsion bath. For example, the emulsion may be poured upon a surface which is desired to be coated and allowed to dry, in most instances in the practice of this invention, at ambient temperatures to form a tough, resilient film on the product. While most of the prior art polyurethane latex films required that a curing, or fusion, step at a temperature of about 150°C. was necessary to form a film having usable properties, we have found that in most instances such a step is not necessary. In using the latex emulsion of this invention to treat textile fabrics, the fabric can be lowered into a pad bath, preferably having about 50% solids, and then removed from the bath and allowed to dry. We have found that when textile fabrics are treated in this manner they become significantly more resistant to abrasion and degradation by scuffing. This is particularly useful in the production of articles such as work clothing and gloves.

The practice of this invention will be further illustrated by the following examples which are offered for purposes of illustration and should not be considered to limit the scope of this invention as described hereinabove.

EXAMPLE I

This example describes the general pro-

emulsifiers

nonionic
cationic
anionic
cationic

cedure by which the latices subsequently described are prepared. A number of parameters (emulsifiers, solvent, total solids, etc.) were fixed so that the effect of other variables—particularly the chain extender used—can be seen.

A prepolymer was made by heating for four hours at 80-90° C. a mixture of a polymeric diol and a 100% excess of a diisocyanate (NCO/OH=2.0/1). To a solution of 140 parts by weight of the urethane prepolymer and 30 parts of toluene was added 7 parts by weight of an emulsifier consisting of dihydric polyoxyethylene polyoxypropylene having a molecular weight of 16,500, a polyoxyethylene content of 80 wt. % and a molecular weight of the polyoxypropylene base of about 3,250 dissolved in 93 parts of water while the former solution was stirred rapidly for two minutes by means of a Premier Dispersator.

After the resulting prepolymer emulsion was passed through a hand-operated homogenizer, an aqueous solution containing one molar equivalent amount of a polyoxypropylenediamine per free isocyanato groups in the prepolymer was added with moderate stirring. The polyoxypropylenediamine solution contained enough water so that the final emulsion, or latex, contained 50% total solids.

After aging the latex for several days, a film 15 mils thick was cast in an aluminum mold. After drying, the film was stripped from the mold and subjected to various tests.

EXAMPLE II

Through the use of polyoxypropylenediamines as chain extenders it is possible to obtain urethane latex films having excellent mechanical properties without having to resort to high-temperature fusion.

A series of prepolymers was made as described in Example I using commercially available 80/20 mixture of 2,4-, 2,6-toluene diisocyanate and polypropylene glycols (PPG's) of molecular weights 600, 800 and 1,200. The polyoxypropylenediamines and 2-methylpiperazine (2-MP) were used as chain extenders to prepare latices in the manner described in Example I. Unfused film chain extended with polyoxypropylenediamine were compared with films derived from 2-methylpiperazine, both unfused and fused at 150° C. for 10 minutes. Results of the comparison are shown in Table I.

When films having the same feel, or "hand", are compared (100% modulus and percent ultimate elongation are indications of the property), those chain extended with polyoxypropylenediamine are stronger than fused films chain extended with 2-methylpiperazine. The film based on 2-methylpiperazine and PPG-600 has superior strength, but this film is very stiff, i.e., poor "hand".

TABLE I

Comparison of Film Properties of Latices Chain-Extended with polyoxypropylenediamine vs. 2-Methylpiperazine (2-MP)

Test No.	Chain Extender	PPG mol. wt.	Fused or unfused	Tensile strength psi.	100% modulus psi.	% Ultimate elongation	Tear initiation lbs./mil	Tear propagation, lbs. Initial	Tear propagation, lbs. Maximum	Abrasion resistance
1	POPDA-190*	600	Unfused	2600	300	600	0.16	0.77	1.36	Fair
2	POPDA-190	800	Unfused	1800	200	700	0.15	0.90	1.30	Good
3	POPDA-240	800	Unfused	1550	300	750	0.15	0.55	0.72	Good
4	POPDA-190	1200	Unfused	800	170	1100	0.10	0.52	0.55	Poor
5	2-MP	600	Unfused	2740	1640	200	0.37	1.15	3.12	Good
6	2-MP	800	Fused	4720	2640	270	0.55	1.86	4.21	Good
			Unfused	1400	620	260	0.03	0.59	0.87	Good
7	2-MP	1200	Fused	2100	360	480	0.13	0.31	2.24	Good
			Unfused	1470	280	600	0.14	0.82	2.46	Good
			Fused	2310	320	1210	0.19	1.35	1.95	Good

*Jefferson Chemical Co., Inc.'s polyoxypropylenediamine. The numerals following indicate molecular weight.

EXAMPLE III

This example illustrates that it is not necessary to limit the prepolymer polyol to those containing branching ($-\text{CH}_2$) when 5 polyoxypropylenediamines are used as chain extenders.

Prepolymers were made as described in Example I from commercial toluene diisocyanate and polytetramethylene ether glycol of 1,000 molecular weight. When 2-methylpiperazine was used as the chain extender, films mud-cracked. Continuous films were obtained when polyoxypropylenediamines of 190 and 400 molecular weights were used as 15 chain extenders.

EXAMPLE IV

Many of the water-soluble diamines that one would consider as chain extenders frequently cause coagulation upon addition to the prepolymer emulsion or give latices 25 the films from which mud-crack. A stable latex is formed and good films result when the prepolymer is chain extended using polyoxypropylenediamine as described in Example I.

Table 2 illustrates the comparison of polyoxypropylenediamine chain extenders with other chain extenders in combination with conventional prepolymers. 30

TABLE 2
Prepolymer Composition

Experiment	Chain Extender	Polyol	Diisocyanate	Results
35	A POPDA-190	PPG-800	TDI	Satisfactory latex & film
	B POPDA-400	"	"	"
	C Ethylenediamine	"	"	Caused "coagulation"
	D 1,3-Propanediamine	"	"	"
	E 1,3-Diamino-2-hydroxypropane	"	"	"
40	F Hexamethylenediamine	"	"	Film mud-cracked
	G Piperazine	"	"	"
	H 2-Methylpiperazine	"	"	Satisfactory latex & film
	I POPDA-190	PEG-PPG-1000*	"	"
45	K 2-MP	"	"	Caused "coagulation"
	L POPDA-190	Polytetramethylene Ether Glycol-1000	"	Satisfactory latex & film
50	M POPDA-400	"	"	"
	N 2-MP	"	"	Mud-cracked
	O 1,3-Propanediamine	"	"	Caused coagulation
	P POPDA-190	PPG-800	Hydrogenated 4,4'-diphenylmethane diisocyanate	Satisfactory latex & film
55	Q POPDA-400	PPG-800	"	"
	R 2-MP	PPG-800	"	Mud-cracked

*A polyoxyethylene poly-1,2-polyoxypropylene glycol of 1000 mol. wt. containing 30% polyoxyethylene units, consisting of a PPG-400 base, followed a polyoxyethylene block, then a poly-1,2-polyoxypropylene block, and finally capped each end with a 5% polyoxyethylene unit. 60

EXAMPLE V

It is not necessary that the polyoxypropylenediamine be completely water-soluble, as this example illustrates. 65

A prepolymer was made as described in Example I from the commercial toluene diisocyanate and polypropylene glycol of 800 molecular weight. A prepolymer emulsion was made and extended with polyoxypropylenediamine of 1,000 molecular weight as described in Example I. Even though the 70 extender was not completely soluble in the aqueous phase of the emulsion, over a period of about 15 minutes the extender dissolved a little at a time and diffused into the suspended prepolymer, where it reacted with 75 the free isocyanate groups. Stirring was continued for 45 minutes to assure complete 80

solution and reaction.

The film cast from the resulting latex was very soft and highly extensible. 85

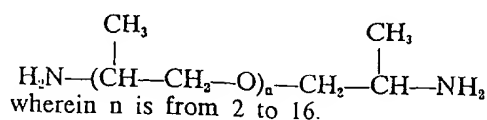
WHAT WE CLAIM IS:—

1. An elastomeric latex comprising an oil-in-water emulsion of the reaction product of:

(a) an isocyanato-terminated prepolymer prepared by reacting a molar excess of an organic diisocyanate and a polymeric diol having a molecular weight of from 400 to 2,000, the molar ratio of the diisocyanate to the polymeric diol being between 1.5:1 and 2.0:1 and, 90

(b) 0.8 to 1.2 molar equivalents of a polyoxypropylenediamine chain extender per molar equivalent of excess isocyanato 95

groups of the prepolymer, said polyoxypropylenediamine having an average molecular weight of from 190 to 1,000 and having the general formula:



2. An elastomeric latex according to claim 1, wherein the polymeric diol is a polyalkylene ether glycol.

3. An elastomeric latex according to claim 2, wherein the polyalkylene ether glycol is polypropylene glycol.

4. An elastomeric latex according to claim 3, wherein the polypropylene glycol has a molecular weight of from 600 to 1,200.

5. An elastomeric latex according to any one of claims 1 to 4, wherein the polyoxypropylenediamine has a molecular weight of from 190 to 500.

6. An elastomeric latex according to any one of claims 1 to 5, wherein about 1.0 equivalent of the polyoxypropylenediamine is present per molar equivalent of excess isocyanate groups in the prepolymer.

7. An elastomeric latex according to claim 1, substantially as herein described.

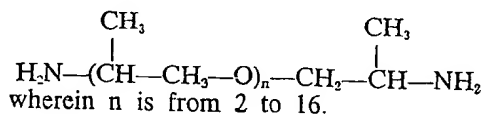
8. A process for the manufacture of stable elastomeric latex which comprises:

(a) reacting a molar excess of an organic diisocyanate with a polymeric diol having a molecular weight of from 400 to 2,000, the molar ratio of the diisocyanate to the polymeric diol being between 1.5:1 and

2.0:1, to form an isocyanato terminated prepolymer;

(b) emulsifying the prepolymer in an aqueous medium with agitation in the presence of an emulsifying agent which will give oil-in-water emulsion, and

(c) chain extending the emulsified prepolymer by adding 0.8 to 1.2 molar equivalents per molar equivalent unreacted isocyanato group of a polyoxypropylenediamine having a molecular weight of from 190 to 1,000 and of the general formula:



9. A process according to claim 9, wherein the polymeric diol is polypropylene glycol having a molecular weight of 600 to 1,200; the organic diisocyanate is toluene diisocyanate; the polyoxypropylene diamine has a molecular weight of 190 to 500 and is added in an amount such that about 1.0 molar equivalent is present per molar equivalent of excess isocyanate groups.

10. A process according to claim 8 and substantially as herein described with reference to the specific Examples.

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